UNIVERSITI PUTRA MALAYSIA

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MASTER OF SCIENCE
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SYNTHESIS OF BISMUTH VANADATE AND COPPER-DOPED BISMUTH VANADATE AS VISIBLE LIGHT PHOTOCATALYSTS

By

NORSALINDA BTE MOHD ALI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

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Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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Chairman: Abdul Halim Abdullah,PhD

Faculty: Science

Since the majority of sunlight consists of visible light and only 3-5% of UV light, numerous studies has been pointed out to develop an alternative semiconductor that have higher efficiency on visible light irradiation. Visible light induced $BiVO_4$ photocatalyst with monoclinic structure has been successfully synthesized via precipitation method. Two different sources of bismuth namely bismuth acetate and bismuth nitrate pentahydrate were used. A solution of bismuth salts were mixed with NH_4VO_3 solution individually before being titrated against NH_4HCO_3 solution. The yellowish precipitated formed was calcined at 450^oC for 4 hours under air flow.

All peaks recorded from XRD pattern, well correspond to monoclinic $BiVO_4$. The FT-IR spectra of the sample powders showed main bands at 800–900 cm^{-1}, assigned as vanadate stretching mode. The scanning electron micrographs showed both $BiVO_4$ were

agglomerated with each other due to the smaller surface area. The BET surface area of $BiVO_4$ produced from bismuth nitrate pentahydrate was higher (3.92 m^2/g) than that produced from bismuth acetate (2.68 m^2/g). Higher surface area (4.29 m^2/g) photocatalyst from bismuth nitrate pentahydrate was obtained when calcination temperature reduced to 300^oC. It has been applied for photodegradation of MB and RO16 under visible light irradiation. The photodegradation carried out at varying catalyst dosage, dye concentration and pH of the dye. The photodegradation of MB was gradually increased with the increasing of $BiVO_4$ loading up to 0.6 gram with 34% removal while for RO16, the optimum loading was 0.8 gram with 95%. In order to extend the efficiency of photodegradation, prepared $BiVO_4$ was modified by introducing Cu via impregnation method. The mixed solutions of $BiVO_4$ and Cu salt solution were heated at 80^oC until all the water evaporated and the calcination process carried out next at 350^oC. The same parameters studied for the photodegradation experiments. It showed that 1% Cu-doped $BiVO_4$ gives higher percentage removal of MB and 3% Cu-doped $BiVO_4$ work best for photodegradation of RO16. Both results revealed that the optimum catalyst loading was 0.5 gram with 36 and 100% removal for MB and RO16, respectively. Cu-doped $BiVO_4$ show surface area at 5.76, 4.04 and 4.28 m^2/g for 1, 3 and 5% Cu-doped $BiVO_4$, respectively.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

SINTESIS DAN PENCIRIAN BISMUT VANADAT DAN KUPRUM-DOP BISMUT VANADAT SEBAGAI FOTOMANGKIN CAHAYA NAMPAK

Oleh

NORSALINDA BTE MOHD ALI

April 2010

Pengerusi: Prof. Madya Dr. Abdul Halim Abdullah

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Oleh kerana kebanyakan cahaya matahari mengandungi cahaya nampak, dan hanya 3-5% sahaja cahaya ultraungu, pelbagai kajian telah dijalankan oleh untuk membangunkan semikonduktor yang mampu memberi reaksi terhadap sinaran cahaya nampak. Mangkin $BiVO_4$ yang berfungsi dalam sinaran cahaya nampak dengan struktur monoklinik, berjaya disediakan melalui pemendakan proses. Dua punca bismut telah digunakan di sini iaitu bismut asetat dan bismut nitrat pentahidrat. Larutan garam bismut dicampurkan dengan larutan ammonium metavanadat terlebih dahulu sebelum dititrat dengan larutan ammonium hidrogen karbonat. Mendakan kuning yang terhasil dipanaskan pada suhu 450^{o}C selama 4 jam dibawah aliran udara termampat.

Kesemua puncak yang direkodkan daripada paten XRD, menurut kepada JCPDS Fail no: 014-688 yang sejajar dengan monoklinik $BiVO_4$. Spektrum FT-IR untuk kedua sampel menunjukkan jalur utama pada 800–900 cm^{-1} yang mana ditetapkan untuk mod regangan

vanadat. Mikroskop Imbasan Elektron menunjukkan kedua-dua sampel beraglomerasi antara satu sama lain disebabkan oleh luas permukaan yang kecil. Luas pemukaan BET untuk $BiVO_4$ yang dihasilkan daripada bismut nitrat pentahidrat adalah lebih tinggi (3.92 m^2/g) berbanding dengan yang dihasilkan daripada bismut asetat
(2.68 m^2/g). Luas permukaan yang lebih besar (4.29 m^2/g) untuk fotomangkin daripada bismut nitrat pentahidrat diperolehi apabila suhu kalsinasi diturunkan kepada 300^oC. Ianya digunakan dalam aplikasi fotodegradasi untuk metilena biru dan reaktif oren 16 dibawah sinaran cahaya nampak. Degradasi cahaya dijalankan berdasarkan pelbagai dos mangkin, kepekatan dan pH pewarna. Fotodegradasi bagi metilena biru meningkat dengan peningkatan dos $BiVO_4$ sehingga 0.6 gram memberikan 34% penyingkiran manakala untuk reaktif oren, optimum muatan adalah 0.8 gram dengan 95%. Untuk meningkatkan kecekapan degradasi cahaya, $BiVO_4$ yang disediakan telah diubahsuai dengan memperkenalkan kuprum melalui cara impregnasi. Campuran larutan $BiVO_4$ dan larutan garam kuprum dipanaskan pada suhu 80^oC sehingga semua kandungan air hilang dan proses kalsinasi dijalankan pada suhu 350^oC. Parameter yang sama dikaji untuk proses degradasi cahaya. Ia menunjukkan bahawa 1% Cu-dop $BiVO_4$ memberikan peratus penyingkiran yang tinggi untuk MB dan 3% Cu-dop $BiVO_4$ berfungsi dengan baik untuk RO16. Kedua-dua analisa menunjukkan bahawa optimum dos pemangkin adalah 0.5 gram dengan 36% dan 100% penyingkiran bagi MB dan RO16. Luas permukaan bagi 1, 3 and 5% Cu-dop $BiVO_4$ adalah 5.76, 4.04 dan 4.28 m^2/g masing-masing.

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This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of Supervisory Committee were as follows:

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

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